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ORIGINAL ARTICLE

Adsorption of imazethapyr on six agricultural soils of Morocco: Evaluation of the impact of soil properties

S. Oufqir *, M. El Madani, M. Alaoui El Belghiti, A. Zrineh, M. El Azzouzi

Structure de Physico-Chimie des Matériaux et Nanomatériaux, Département de Chimie, Faculté des sciences, Université Mohammed V-Agdal, BP 1014-Avenue Ibn Battouta, Rabat, Morocco

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KEYWORDS

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Abstract Imazethapyr herbicide [5-ethyl-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl) nicotinic acid] is widely used on agricultural field crops in Morocco. It is reported that soil properties largely enhance the sorption of the herbicide in soils, however understanding their impact in soils of Morocco is needed. The present article investigates the sorption behavior of imazethapyr herbicide toward six agricultural soils from Morocco in batch equilibrium experiments at pH 5 and evaluates the effect of the organic matter, the clay content, and the cation exchange capacity (CEC) on the sorption by applying simple and multiple linear regressions. Freundlich isotherm model described well the dynamic of imazethapyr sorption in all studied soils under the experimental conditions. The linear regressions showed significant differences between the soil properties and their impact on the sorption. While simple regression revealed strong positive correlations between the sorption and both the soil clay content (R^2 = 0.91) and CEC (R^2 = 0.92), the multiple regression revealed a noteworthy influence of soil organic matter when combined with clays. Our findings showed a weak adsorption of imazethapyr on the selected Moroccan soils, in addition to a potential role of clay minerals and CEC in the retention of the herbicide compared to the organic matter. The low adsorptive capacity of the Moroccan soils urges the necessity of minimizing herbicide application rates in agricultural regions to prevent environmental detrimental impacts.

1. Introduction

Imazethapyr [5-ethyl-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl) nicotinic acid] belongs to the Imidazolinone family of herbicides that become extensively used in a wide range of cropping systems in Morocco because of their low application rates and reduced selectivity. However, they are considered priority pollutants in soil. To better understand the behavior and mobility of imazethapyr in the Moroccan environment and avoid their harmful effects on target and non-target

* Corresponding author. Tel.: +212 668699115.
E-mail address: soufqir@yahoo.fr (S. Oufqir).

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organisms, it is essential to consider its adsorption capacity which represents the most efficient and promising process determining the fate of herbicides in the environment (Chefetz et al., 2004; Gupta et al., 2009, 2011). A breadth of knowledge on the sorption of herbicides in a variety of terrestrial soils exists, but information on imazethapyr in soils of Morocco is lacking.

The imazethapyr molecule is amphoteric of nature carrying both acidic (carboxyl) and basic (pyridine nitrogen) functional groups. Its ionic state depends upon the pH conditions of the medium: at neutral or high pH, the anionic form predominates causing very low or negative adsorption of the herbicide due to the repulsion of the anions from the negatively charged clay and organic surfaces (Green, 1974; Weed and Weber, 1974; Renner et al., 1988; Loux et al., 1989; Stougaard et al., 1990; Nègre et al., 2001; Bresnahan et al., 2002); at lower pH, a nonionic form of the herbicide prevails promoting high affinity for soil typical of many nonionic herbicides (Singh et al., 1990; Stougaard et al., 1990; Che et al., 1992; Loux and Reese, 1993; Gennari et al., 1998; El Madani et al., 2003). Besides molecular structure, various soil constituents and properties, such as organic matter, clay content, and cation exchange capacity (CEC) affect the persistence of imazethapyr in soil and its adsorption magnitude at low pH (Renner et al., 1988; Loux et al., 1989; Stougaard et al., 1990; Goetz et al., 1990; Curran et al., 1992; Gennari et al., 1998; Jourdan et al., 1998; Wang and Lin, 1999; Johnson et al., 2000). However, the literature is divided based on which characteristic controls primarily the sorption in soils from different geographical origins. Substantial body of research indicated that soil organic matter has high affinity for imazethapyr (Stougaard et al., 1990; Che et al., 1992; Senesi et al., 1997; Gennari et al., 1998; Nègre et al., 2001), while others stated that the organic matter content may not entirely account for the adsorption of imazethapyr by soil particles (Ahmad et al., 2001; Oliveira et al., 2001; Sakaliene et al., 2007; Ahmad and Rahman, 2009). Likewise, conflicting results have been reported regarding the role of soil clay content and CEC in the adsorption; no effect of clay minerals and CEC on the sorption (Ahmad et al., 2001; Leone et al., 2002; Cruz-Guzman et al., 2005; Ahmad and Rahman, 2009) versus positive impact in soils (Loux et al., 1989; Stougaard et al., 1990; Che et al., 1992; Sakaliene et al., 2007).

In aggregate, studies conducted on the behavior of imazethapyr in soil have yielded variable results depending upon climatic and environmental contexts. But to date only studies on the effect of pH have been published in the Moroccan context (El Madani et al., 2003). In this study we seek to obtain information about the sorption of imazethapyr and the factors influencing it to better determine pesticide exposures and threats, and to develop efficient management and remediation approaches where necessary. Our objectives were to investigate the adsorption capacity of imazethapyr in six soils from different agricultural regions of Morocco at pH 5 using adsorption isotherms and to evaluate the effect of cation exchange capacity (CEC), organic matter content, and clay content on the sorption process by means of statistical methods.

2. Materials and methods

2.1. Herbicide

Imazethapyr herbicide [(RS)-5-ethyl-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl) nicotinic acid] of 99.9% purity was purchased from the Riedel de Haën company, Seelze (Germany). Imazethapyr is a weak acid with pKa values of pKa$_1$ = 2.1 and pKa$_2$ = 3.9 (Worthing and Hance, 1991). The physical and chemical properties of the herbicide are given in Table 1, and the structures of its ionic forms are shown in Fig. 1.

2.2. Soil samples

Six soil samples were collected from six agricultural fields in different regions of Morocco at 0–15 cm depth. All samples were air-dried at room temperature, carefully homogenized, sieved to a particle size $\leqslant$2 mm, and then stored without any pretreatment. The selection of the soils was based on the representation of a wide range of characteristics; organic matter content from 0.38% to 9.34%, clay content from 7.80% to 34.80% with smectite being the dominant clay mineral in all soils, and CEC ranging from 21 to 63 cmol/kg sol (Table 2). The organic matter, the CEC, and the clay content were determined using the chromic acid digestion method (Walkley and Black, 1934), the ammonium acetate method (Chapman, 1965), and the hydrometer method (Gee and Bauder, 1986) respectively. The clay mineralogy of the soil samples was determined using X-ray powder diffraction on oriented thin clay film samples.

Table 1 Physical and chemical properties of imazethapyr herbicide: Molecular weight, Water solubility, Octanol–Water partition coefficient (K_{ow}), Melting point, and Specific gravity.

Herbicide	Molecular weight	Water solubility	K_{ow}	Melting oint	Specific gravity
Imazethapyr	289.34 g/mole	1.4 g/l (25 °C)	11 (pH 5), 31 (pH 7)	169–173 °C	1.10–1.12 g/ml (21 °C)

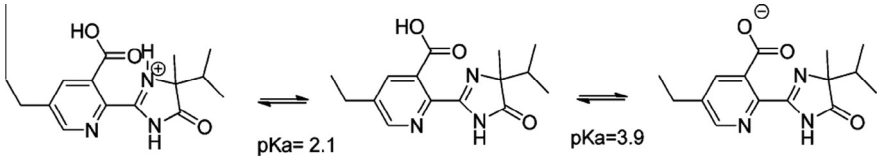

Figure 1 Chemical structure of imazethapyr herbicide; neutral state; cationic state and anionic state.

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Table 2 Freundlich adsorption constants for imazethapyr sorption and organic matter content, texture data, original pH values, and CEC of the investigated soils. Collected from different regions, Gharb East, Gharb West, Ghard North, Chaouia East, Chaouia West, and Chaouia North, and the soil type is Vertisol, Sandy, Cambisol, Luvisol, Calcisol, and Calsisol, respectively.

	Soils					
	1	2	3	4	5	6
Geographical site	Gharb East	Gharb West	Gharb North	Chaouia East	Chaoui West	Chaoui North
Soil type	Vertisol	Sandy	Cambisol	Luvisol	Calcisol	Calsisol
Soil characteristics						
OM*,%	2.68	0.38	9.34	6.47	5.2	1.55
Clay,%	34.8	7.8	10	20.2	9.8	20.7
Silt,%	30.6	22.9	55	43	50	46
Sand,%	34.6	69.3	35	36.8	40.2	33.3
pH, H_2O	8.38	8.1	7.49	7.71	7.9	7.84
CEC (cmol/kg)	63	21	35	29	33	41
K_f	37.8	3.9	114.4	9.5	4.1	17.2

* OM: organic matter content.

2.3. Adsorption experiments

A batch equilibrium method adsorption chromatography was used to determine the sorption of imazethapyr herbicide by the selected soil samples at room temperature employing 0.01 M $CaCl_2$ as a background electrolyte. Adsorption isotherms were performed in duplicate using 17.28, 34.57, 51.85, 69.13 and 85.41 μmol/L concentrations of imazethapyr water solutions prepared from a stock solution of 1.73×10^3 μmol/L. The pH was adjusted to pH 5 ± 0.2 using 1 M H_2SO_4/KOH, at which the non-ionized form is predominant in solution promoting the interaction with negatively charged soil colloids (El Madani et al., 2003). 2 g of air-dried soil was shaken with 10 mL of each imazethapyr solution (10 mg/L in 0.01 M $CaCl_2$) for 16 h at 200 rpm in centrifuge glass tubes sealed with specially designed PTFE caps and covered with a thin aluminum foil in order to prevent losses by adsorption on the caps. Preliminary experiments have shown that equilibrium is reached within 12 h and remains stable for 24 h. Soil-Imazethapyr solutions were centrifuged at 3000 rpm for 25 min to precipitate soil particles larger than 0.2 μm. The supernatant was filtered using a glass fiber filter, and stored at 4 °C for further analyses.

Concentrations of free imazethapyr in the supernatant were measured by high pressure liquid chromatography (HPLC) using a Perkin Elmer Series 2 Liquid Chromatograph equipped with UV detector, and a C18 Luna column of 4 mm diameter and 250 mm lengths packed with 5 μm size particles. The mobile phase was an isocratic solution of 60% (v/v) water and 40% of an aqueous solution of acetonitrile with pH set up at 2 using H_3PO_4 acid. The flow rate was 1 mL min^{-1} and the injection volume was 50 μl. Detection was at about 255 nm. Under these conditions, the retention time for imazethapyr was 5.65 min. Its calibration was calculated on the basis of the peak areas obtained with standardized samples analyzed under the same HPLC conditions. Control blanks of the system soil with $CaCl_2$ and of the test substance with no soil were run in parallel at each imazethapyr concentration used in the experiments to check for matrix effects caused by the soil and the degradation of imazethapyr.

Adsorption data were fit to the Freundlich equation $Y = K_f C_s^{nf}$ (Sposito, 1989), where Y is the amount adsorbed on soil at adsorption equilibrium in μmol kg^{-1}, C_s is the equilibrium solution concentration of imazethapyr in μmol L^{-1}, the constant K_f is a measure of the adsorption capacity of the sorbent, and the constant nf indicates the degree of nonlinearity of the sorption data. Freundlich K_f values and nf values were computed from the logarithmic form of the equation $\log Y = \log K_f + nf \log C_s$.

2.4. Statistical analysis

Statistical analysis was performed using R Statistical Software, a free software environment for statistical computing and graphics, to assess the effect of the soil properties on the sorption of imazethapyr. Accordingly, simple and multiple linear regressions were applied to K_f values and three soil parameters namely, organic matter content, clay content, and CEC. With a relatively higher amount of organic matter (9%) in soil 3, calculation showed that 9% was an outlier value, and hence, soil 3 was excluded from the analysis in order to meet linear regression assumptions.

3. Results and discussions

3.1. Sorption of imazethapyr

The adsorption capabilities of six agricultural soils toward imazethapyr herbicide were investigated at pH 5. Experimental data depicted in Fig. 2 were successfully described by Freundlich isotherms over the concentration range tested. The computation of adsorption coefficients K_f produced low values ranging from 3.9×10^{-5} to 114.4×10^{-5} (Table 2) which indicates a weak sorption of imazethapyr by soil particles. This reflects high persistence for a long time in soils (Hollaway et al., 2006; Cessna et al., 2012), that can increase the potential carry over damage to non-target crops in rotations in subsequent years (Onofri, 1996). Moreover, it insinuates the contamination of the ground water through leaching process in a country experiencing physical water scarcity, and hence creates a threat to drinking water (Dabrowski et al., 2002).

Compared to the studied soils, soil 1 and soil 3 showed a high sorption potential for imazethapyr as well as a high degree of linearity indicating a great homogeneity in their

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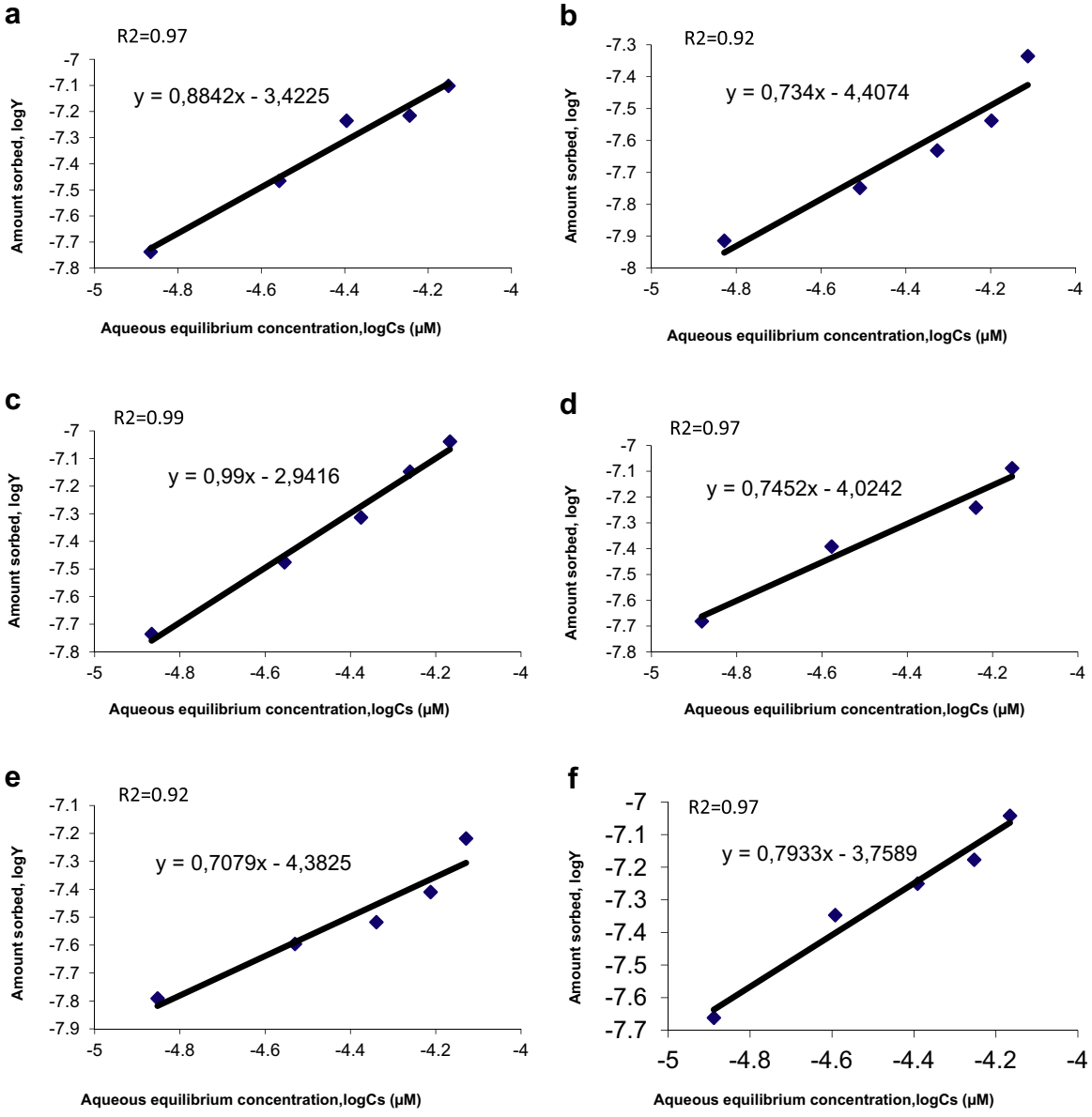

Figure 2 Measured linearization of Freundlich sorption isotherms for Imazethapyr herbicide to soil 1 Gharb East (a), soil 2 Gharb West (b), soil 3 Gharb North (c), soil 4 Chaouia East (d), soil 5 Chaouia West, and soil 6 Chaouia North (f).

binding sites (Table 2). In contrast, soil 2 and soil 5, with similar nf values, revealed a poor sorption although the amount of organic matter contained in soil 5 was significantly bigger than in soil 1. On the other hand, the sorption on soils 4 and 6, with comparable clay content and distinct organic matter content, resulted in a similar shape of isotherms. From these data, we can infer that the negatively charged smectite particles, that have a fairly high CEC charge (Marshall, 1935), greatly enhance the uptake of the molecular form of imazethapyr compared to the organic matter content within the same soil. The smectitic clay minerals and the CEC seem then to be the main factors controlling the sorption. The organic matter might have no influence on the adsorption, which is in agreement with the results of Oliveira et al. (2001) and Sakaliene et al. (2007). The substantial variability in the behavior of the six soils toward imazethapyr sorption, sustained by the differences in nf values, implies that the soil properties affect the sorption. In order to understand properly which soil property influences the sorption of imazethapyr in soils of Morocco, simple and multiple linear regressions were conducted.

3.2. *Effect of soil properties on the sorption of imazethapyr*

Statistical correlations between Freundlich constant K_f and the soil characteristics namely organic matter content, clay content, and CEC have been carried out for the soils 1, 2, 4, 5, and 6. Simple linear regression results revealed that imazethapyr adsorption has a significantly strong positive correlation with both clay minerals and CEC (Fig. 3 and 4), but no correlation with organic matter content at $p < 0.05$ for which data are not shown in this paper. This suggests that the sorption is favored by CEC and clay minerals and disfavored by organic matter content in the studied set of soils.

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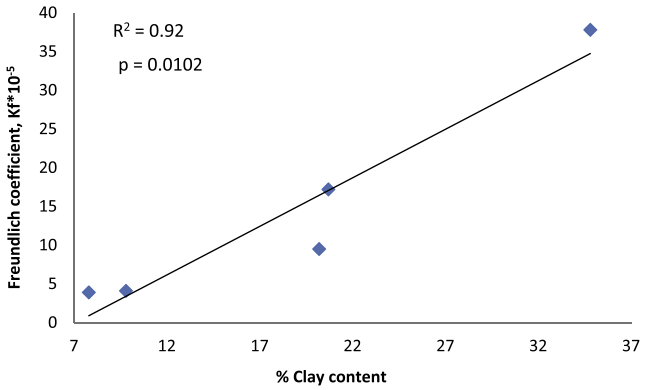

Figure 3 Effect of clay content on the adsorption of imazethapyr on soils of Morocco established by linear regression between K_f and soil clay content.

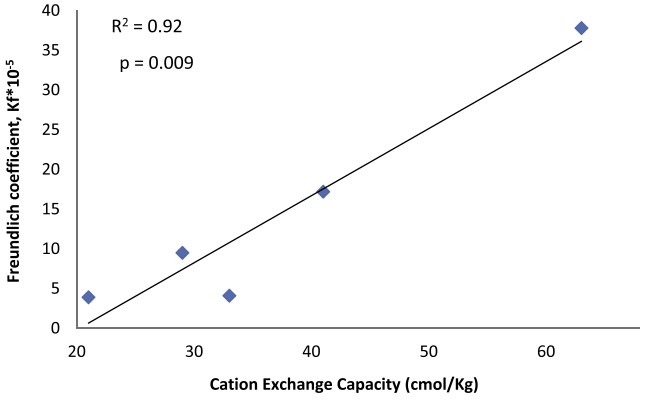

Figure 4 Effect of CEC on the adsorption of imazathapyr on soils of Morocco established by linear regression between K_f and CEC.

Interestingly, clay minerals explained 91.57% ($p = 0.01$) of the variation of the sorption while CEC explained 92.14% ($p = 0.009$). This confirms them being the major predictors describing the sorption of imazethapyr, and supports the findings of Loux et al. (1989), Stougaard et al. (1990), Che et al. (1992), and Sakaliene et al. (2007), but opposes the conclusion of Leone et al. (2002) about the inability of smectite to adsorb the herbicide.

It is noteworthy that the CEC correlated strongly with the smectite particles as compared to the organic matter, and that the inclusion of the organic matter property to the CEC decreased the correlation coefficient from 0.92 to the level of 0.86 ($p = 0.06$) meaning that the CEC is mainly due to the clay mineral charges. On the other hand, the multiple regression analysis incorporating the combination of clay minerals and organic matter properties substantially strengthened the correlation to $R^2 = 0.93$ ($p = 0.03$) indicating that organic matter might have an effect on the sorption when combined with clay minerals. This inference corroborates with results from previous studies (Ahmad et al., 2001; Ahmad and Rahman, 2009; Sakaliene et al., 2007). The role of organic matter content in controlling the sorption of imazethapyr in Moroccan soils could not be conclusive within the set of soils used in this study. Therefore, further research is needed to investigate the herbicide fate in a wider sample size of soils from various agricultural fields with diverse characteristics representing a homogeneous continuum in order to evidence and discern properly the impact of organic matter and humic-clay composites on the sorption of imazethapyr in soils of Morocco.

4. Conclusion

This study provides preliminary information regarding the sorption of imazethapyr herbicide and the factors controlling this sorption in a selected set of soils of Morocco. We conclude that:

i. Freundlich equation fitted very well the equilibrium data over the entire concentration range studied for the adsorption of imazethapyr with an increasing affinity for soils in this order: Soil 3 > Soil 1 > Soil 6 > Soil 4 > Soil 5 > Soil 2.
ii. All the examined soils showed a weak adsorption capacity for the herbicide.
iii. The sorption of imazethapyr was primarily dependent of both smectitic clay minerals and CEC.
iv. Organic matter content had no effect on the sorption but yielded a slightly strengthened correlation when combined with smectitic minerals.

The organic matter in these soils may have differing sorptivities to imazethapyr herbicide, or may influence its uptake but this could not be evidenced within the set of soils used in this study. Consequently, an extensive investigation of a wide range of soils with variable organic matter content is needed in order to elucidate more properly the role of organic matter in the retention of imazethapyr on agricultural soils in Morocco.

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